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Complicated Conformational Exchange of New Pyridoxine Derivative. Dynamic ^{13}C NMR Characterization

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Abstract New pyridoxine derivative with multiple chemical exchanges was studied by ^{13}C (at 298 K, 188 K) and 2D NMR (^{1}H-^{13}C HSQC, ^{1}H-^{13}C HMBC) spectroscopy in acetone-d_6 solution. Complete ^{13}C NMR data (table with chemical shifts at different temperatures) is presented. Intramolecular mobility of the molecule was analyzed based on the results of ^{13}C NMR experiments in combination with the data obtained from previous study of this compound by dynamic ^{1}H NMR spectroscopy.

Keywords Pyridoxine · ^{13}C NMR spectroscopy · Conformation · Stereochemistry

1 Introduction

Nuclear magnetic resonance (NMR) methods with variation of temperature including the full line shape analysis are very effective for obtaining detailed information about the main characteristics of stereochemically and structurally flexible molecules [1–3]. Nowadays, NMR techniques are also proved to be a powerful tool for conformational analysis of biologically important samples [4–6].

Investigations of systems with multi-rate chemical exchange are primarily relevant because they allow deepening and broadening of our understanding of the mechanism of molecular transformations. Many difficulties might occur related to the assessment of the rate constants and interpretation of activation and kinetic characteristics of the observed processes in study of systems with two or more exchange rate constants (multi-exchange) in combination with more difficult task of components identification [7]. In our previous works, we have studied some pyridoxine derivatives by ^{1}H dynamic NMR spectroscopy [1, 2, 8]. However, ^{13}C NMR spectroscopy also can provide valuable information about this system. In the presented letter, the results of ^{13}C NMR study of new pyridoxine derivative representing a potential biologically active substance and nonlinear optical material – 9-(2,4-dinitrophenoxy)-3,3,8-trimethyl-6-(2-nitrophenyl)-1,5-dihydro-[1, 3] dioxepino[5,6-c]pyridine [9, 10] is discussed. Molecular structure of the compound with possible intramolecular conformational processes (R, R' – rotations, T-T – twist-twist transitions of the cycle) is shown in the top of the Fig. 1.

2 Material and Methods

Registration of 1D (^{13}C) and 2D (^{1}H-^{13}C) NMR spectra of studied compound in $(CD_3)_2CO$ was carried out using NMR spectrometer (Bruker, Avance II-500) (125.76 MHz (^{13}C)) at 298 and 188 K. Temperature control was achieved using a Bruker variable temperature unit (BVT 3000) in combination with a Bruker cooling unit (BCU-05). The sample was cooled by a flow of low-temperature nitrogen gas from a Dewar with liquid nitrogen. The experiments were performed without sample spinning. All two-dimensional experiments were performed with 2 k × 512 data points; the number of transients (2–16 scans) and the sweep widths were optimized individually. All samples were prepared in standard 5-mm NMR tubes. Concentrations of the substances were 0.5 wt%. The solution volume was 0.6 ml. The deuterium signals of the solvent were used for the stabilization of magnetic field.

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